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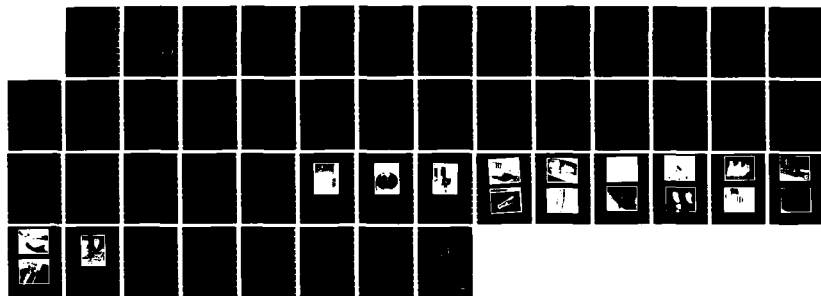
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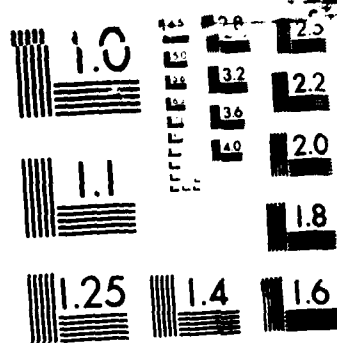
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BURTON TECHNOLOGIES INC
P.O. Box 58088
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CONTROL OF SURFACE ATTACK BY GALLIUM ALLOYS IN ELECTRICAL
CONTACTS

Final Report

submitted to

Office of Naval Research

Contract No. N00014-85-C-0893

Period Covered October 1, 1985 to March 31, 1986

by

Dr. Ralph A. Burton

and

Mr. Gaines Burton

Burton Technologies Incorporated

4940 B. North Boulevard

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March 1986

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INTRODUCTION

The general objective of this Phase I SBIR Program has been to determine the feasibility in going from early NASA work on Ga/In/Sn as contact lubricants to applications in current collectors or brushes where current, duration of operation, and sliding conditions may be severe. This problem involves several variables with complex interactions. These include conduction by the liquid metal, oxidation of the substrate to produce insulating films, the formation and removal of organic contaminant films, the mechanical behavior of the alloy including hydrodynamic support of the contacts, and its being scraped or slung off of the slip rings.

With the long range applications in mind, the program addressed all of these issues to some extent, while developing a test method. The tests reported here cover three widely different kinds of current collectors:

1. Carbon blocks, both as a reference material for apparatus shakedown, and to provide needed data to complete the theory for modeling high performance monolithic current collectors.
2. Porous, compliant quasimonolithic crushed mesh, with the objective of providing a conformally contacting material as well as a source of supply of alloy lubricant held by capillary action.
3. Hydrodynamically acting fingers on an alloy coated slip

ring. These gave the best performance of all and call for extended experiments with controlled oxidation and controlled mechanical management of the lubricant.

For this research two apparatuses were developed. The principal one allows control of atmosphere, observation of the contacts while running, and simultaneous measurement of current, friction, load, contact voltage drop, and film appearance.

BACKGROUND

Compatibility

Gallium is a bright metal with melting point 29.78°C , specific gravity 5.904 (solid) 6.095 (liquid), valence 2 or 3. Unlike the other metals that are liquid near room temperature (mercury, cesium, rubidium) its toxicity is of low order. It alloys with most metals, and as an alloying ingredient aids metals such as tin in wetting nickel and stainless steel. Gallium has a viscosity of 1.89 cp at 50°C , and an electrical resistance of 17.4 micro-ohms cm. compared to 800 micro-ohms cm for brush carbon, and 1.7 micro-ohm cm for copper at room temperature. Melting point surface tension is 704 dynes/cm, which is comparable to 542 for Hg. When pure gallium is cooled, it may remain liquid to temperatures as low as -4°C .

Table I shows the position of gallium in the electromotive series, along with selected structural and alloying metals. Along with other data this may give indications as to compatibility behavior.

Buckley and Johnson (1) drawing upon (2,3,4) indicate compatibility between gallium and several structural materials, as summarized in Table II.

TABLE I
OXIDATION-REDUCTION EQUILIBRIUM CONSTANTS FOR SOME METALS

| HALF REACTION | CONSTANT |
|---|--------------|
| $K \rightarrow K^+$ | $10^{49.5}$ |
| $Na \rightarrow Na^+$ | $10^{46.0}$ |
| $Mg \rightarrow Mg^{++}$ | $10^{40.6}$ |
| $Al \rightarrow Al^{+++}$ | $10^{28.8}$ |
| $Mn \rightarrow Mn^{++}$ | $10^{18.6}$ |
| $Zn \rightarrow Zn^{++}$ | $10^{12.8}$ |
| $Cr \rightarrow Cr^{++}$ | $10^{10.2}$ |
| <u>$Ga \rightarrow Ga^{+++}$</u> | $10^{8.5}$ |
| $Fe \rightarrow Fe^{++}$ | $10^{7.4}$ |
| $Cr^{++} \rightarrow Cr^{+++}$ | $10^{6.8}$ |
| $Cd \rightarrow Cd^{++}$ | $10^{6.7}$ |
| $In \rightarrow In^{+++}$ | $10^{6.4}$ |
| $Ti^{++} \rightarrow Ti^{+++}$ | $10^{6.3}$ |
| $Co \rightarrow Co^{++}$ | $10^{4.9}$ |
| $Ni \rightarrow Ni^{++}$ | $10^{3.7}$ |
| $Sn \rightarrow Sn^{++}$ | $10^{2.2}$ |
| $Pb \rightarrow Pb^{++}$ | $10^{2.0}$ |
| $Cu^+ \rightarrow Cu^{++}$ | $10^{-2.9}$ |
| $Au \rightarrow Au^+$ | $10^{-25.4}$ |

This data compiled from **Qualitative Analysis and Chemical Equilibrium**, Hogness and Johnson, published by Henry Holt and Co. of New York.

TABLE II. COMPATIBILITY OF LIQUID GALLIUM WITH SOLID MATERIALS BELOW 400°F

| Compatibility | Materials |
|---------------|--|
| Good | 18-8 Stainless Steel, 16 Cr Stainless Steel, Tantalum 95 Te-7W, Niobium, Molybdenum Molybdenum, Titanium, Lead Carbon, BeO, Quartz, Al ₂ O ₃ , ZrO, Pyrex glass |
| Poor | Iron, Aluminum, Silver, Tin Zinc |
| Unknown | Tool Steel, Chromium, Manganese, Nickel, Copper, Platinum |

Kuczkowski and Buckley (2) showed nickel and 440C stainless steel to perform well with gallium alloys in the presence of abrasion.

A recent study (5) reports results for structural materials and polymers in Ga 67/In 20.5/Sn 12.5, an eutectic which is close to the alloy found most promising as a lubricant in Ref. (7). Samples were immersed for 1000 to 1500 hours. Suitable metals were found to be: Tantalum, Copper and Bronze, Vanadium and Titanium. Because of the attractiveness of copper in electrical systems and bronze in

bearing systems, we were encouraged to look further into the use of copper.

Both iron and aluminum were rated poor in Table I. Our colleague, Dr. H. Conrad warned us about grain boundary attack of aluminum, but he showed little concern regarding iron alloys. In our Phase I experience iron (mild steel) has shown no observable problems, but aluminum has shown a singular sensitivity. Trace amounts of gallium alloy on the surface of 0.25 in. thick aluminum structural members led to complete embrittlement, explosive fracture and distortion along with discoloration of the fractured surfaces. The members so attacked had been cold worked. One reference to such behavior was found for a close relative to aluminum, zinc (6). This reference documents the rapid diffusion of gallium indium melts along the grain boundaries of polycrystalline zinc, and the presence of liquid in these regions.

Heat Treatment of Gallium-alloy Films

Buckley and Johnson (1) mention heat treatment of applied films of gallium alloys to produce a solid film which would melt under frictional heating. Favorable boundary lubrication was provided by such films. Some insight is provided by reference to binary phase diagrams for gallium with the following materials: gold, cobalt, chromium, copper, iron, molybdenum, niobium, nickel. All of these showed a similar configuration at the high-gallium end. If the gallium is placed on the solid substrate and brought to an appropriate temperature (1000 °F for nickel), then held until equilibrium is established, it will cool to form a solid made up of two intermetallic compounds. If, instead, it is brought to a somewhat lower temperature it will cool to a mixture of liquid and solid, where the ratio of these is dependent upon the temperature. It appears that this latter state was achieved in the reported work. This suggests that fruitful work may be done on surface preparation by heat-treatment of an initial film.

A word of caution on heat treatment techniques has been provided by Cochran and Foster who note that although gallium has a very high boiling point (2676°K), the suboxide Ga_2O has a vapor pressure of 2.4×10^{-6} Torr at 1000°F (537°C). Consequently gallium may in effect sublime in oxidizing atmosphere.

Wetting

Gallium is considered to show exceptional characteristics in wetting metals and some glasses and ceramics. The mechanism by which this occurs is not completely clear. L. M. Foster in discussion of Ref. (1) states unequivocally that "Gallium does not wet surfaces. It is the oxide interface that adheres and gives the appearance of wetting. Gallium from which oxide has been completely removed in vacuum via reaction de-wets the surface and pulls into a ball." In both Ref. (1) and (7) oblique reference is made to the role of oxides in the wetting process.

Our observations in Phase I research have been as follows:

- (1) Gallium actively wet clean copper with thin (estimated at 100 angstrom) CuO film.
- (2) With mild mechanical working Ga/In/Sn alloy wet and covered copper plated steel with oxide film estimated to be CuO_2 at thickness above 300 angstroms.
- (3) Gallium alloy quickly wet and spread on the solid film resulting from friction tests with gallium wetted copper brushes.
- (4) Gallium spread on carbon coated running tracks used with carbon brushes, and adhered to brush faces in a thin film. It was not repelled from the thin copper-carbon bounded brush interface.

(5) Gallium wet a number of organic materials, and adhered to a plastic spatula, a "formica" laboratory bench, and silk-screen applied logo on a laboratory furnace. It adhered to most enameled surfaces it contacted.

(6) No success was found in wetting "feltmetal" nickel or stainless steel as provided by the manufacturer, or on crushed stainless steel mesh.

(7) Brass was actively wetted by the alloy

(8) a pure nickel crucible was wetted, and spreading was observed.

(9) Gallium flung off from the wear track adhered to the glass cover, and to a cardboard barrier.

APPARATUS AND MATERIALS

Equipment And Materials

B & K Precision Oscilloscope Model 1477 with 10X probes (2)

Dayton Model 2Z846A 3/4 HP variable speed constant torque motor

Bently Nevada Series 7200 Proximeter system with 3 probes

Commodore 64 computer system with Micro R & D Company MW-611 input/output board.

Westinghouse industrial micarta grade CE 3/4 inch sheet

Westinghouse industrial micarta grade CE 1 3/4 inch rod

Dayton Model 3Z561A AC Arc Welder

Black and Decker Professional 10 inch circular saw blades

Radio Shack Desoldering Braid

Alfa Thiokol Gallium 99.99% purity

Alfa Thiokol Indium 99.999% purity

Alfa Thiokol Tin 99.5% purity

Brunswick Corporation Nickel Feltmetal Grades FM-1208 and FM-1210

Stock Drive Products crushed stainless steel mesh

Ease Inc. aluminum extrusions

K & S Engineering Co. copper sheet .025 inch

Black and Decker 2 1/2 HP variable speed motor

Structural Graphite supplied by NASA Lewis Research Center

Apparatus

The initial test rig, (Figs. 1,2,3) was constructed to facilitate visual as well as instrumented observation of the experiments. This rig was a turntable type of apparatus constructed from the variable speed motor and a structural micarta board, arranged with the motor shaft passing vertically through the supporting table top and attached to the micarta board by means of the motor's integral mounting flange. On this shaft a mounting hub constructed from the micarta rod was pressed and the specimens were mounted to this hub. The test specimens consisted of a circular saw blades which had been electroplated with copper. The saw blades were chosen because they were precision ground, of a practical diameter and stiffness, and eliminated the excessive cost of machine shop fabrication of what were consumable test specimens. The brushes were mounted on two arms fabricated from the aluminum extrusions and attached to

a mounting post fabricated from the structural micarta sheet by means of a nylon bolt which also served to attach the power leads to these arms. The brushes were attached to the other end of the arms by steel bolts. The brushes were loaded by a spring dynamometer using steel cable. A second mounting post adjacent to the brush end of the arms was attached to an aluminum bar and a proximeter. A steel cable was attached between the aluminum bar and the brush end of the arm assembly to restrain the it, the proximeter read of this aluminum bar to provide the friction force measurement. Voltage drop across the brushes was measured on the oscilloscope using a lead from each brush, (Fig. 4). Power was delivered from the arc welder to the arm assembly and through one brush to the rotating specimen and out the other brush. A glass containment was placed over the test assembly to provide atmospheric control, containment and full visual observation. This arm assembly was found to have the right combination of stiffness and compliance to allow satisfactory brush operation over a very wide range of friction loads without chatter, bounce or galling. Brushes were constructed from the structural graphite , the desoldering braid, and the copper sheet. The graphite brushes were run

both with and without the gallium alloy. The graphite brushes were monolithic. The desoldering braid was a very fine soft copper braid that was found to wet very easily with the gallium alloy and had sufficient compliance to make good contact with the alloy wetted surface. The copper sheet was cut into thin strips three of which were stacked together and bent over to form fingers. These brushes are shown in the accompanying illustrations (figs.10,11,12,13) in their condition after testing. The braid and finger brushes were run only with gallium alloy and were designed solely to provide a vehicle for using the alloy.

A second test apparatus has been designed, constructed, and run but not with the gallium alloy brushes. This rig was designed for long duration testing of the alloy wetted brushes. Its design provides easy containment and atmospheric control but does not allow visual observation of the contact brushes. This machine is a small homopolar motor built from mild steel with ceramic permanent magnets and a central brass rotor. At the present time this is used as a generator and is driven by the Black and Decker variable speed motor, (Fig. 6). The computerized data acquisition

system is being installed with this apparatus to allow constant monitoring and control during extended test runs. This system is capable of reading data and outputting control signals at 100 cycles per second, (Fig. 14). The generator is capable of being sealed to provide self contained controlled atmosphere.

Experimental Procedures

Gallium/indium/tin alloy in the weight percent ratios of 60/20/20 were prepared by mixing the three components together in their respective ammounts and melting the mixture while stirring in a nickel crucible. Heat was applied intermittantly during the melting while the stirring was constant. The alloy formed easily and was observed to be a bright silver color and composed of two phases.

These two phases were a bright silver liquid and a duller silver semisolid. The alloy was applied directly to the copper plated saw blades and smeared with a plastic spatula until a uniform film covered the brush track, (Fig. 18).

During the application of the alloy the brushes themselves were rocked back and forth in the alloy track to wet them and to assist in the spreading of the alloy. The alloy was observed to wet the copper surfaces easily in spite of the presence of a thick oxide layer on the copper.

After the alloy brush track had been applied the glass container was placed over the rig and the turntable was started. Monolithic carbon was run in air and argon atmosphere. Monolithic carbon runs in air were performed both with and without the gallium alloy in the brush track. Copper brushes were prepared from soft copper braid and nested fingers cut from copper strips.

The actual test runs were run until arcing and contact resistance voltages indicated serious deterioration of the contact. The test runs in laboratory air indicated that the gallium alloy lost effectiveness after some time under current load. When the brush track was examined it was found that the bright liquid phase of the alloy was no longer present and the track was a dull silver color much like the semisolid phase of the alloy at preparation. The brush track was easily restored to its original effectiveness by

application of the liquid-phase alloy introduced via a syringe, (Fig. 7). This could be repeated several times with full restoration of the surface upon each reintroduction of fresh alloy. Those runs in argon atmosphere indicated no deterioration of the contact during the runs and current density could be increased to the maximum available from our power source, which was above the indication range of our instrumentation. This run was terminated when the nylon support bolt melted due to the heat generated by the current, there was no decrease in the measured performance of the brush. During all of the runs the liquid alloy was observed to sling off of the specimen (Fig. 15); however those runs in argon retained the bright liquid appearance of the brush track, (Fig. 8) while those in air dulled and required replenishment of the liquid phase. Figure 9 shows the pitting and damage to the wear track from the destruction of the aluminum brush holders from the formation of the gallium aluminum eutectic phase during the test runs with the copper braid these brush holders were replaced by mild steel. All future brush holders designed for use in the presence of the gallium alloy were fabricated from the mild steel. Figures 16 and 17 show both the leading edge of the monolithic

graphite brush with the liquid alloy pooling in front of it, and the clean track at the trailing edge of the brush. Figure 14 is a comparison photograph of the condition of the unused graphite face, the graphite-on-copper wear face and the graphite-on-gallium alloy wear face. The graphite on gallium wear face shows the wetting of the graphite by the gallium alloy and less evidence of abrasion than the gallium on copper. It was further noted that the copper specimens wetted more easily when a graphite residue was present in the brush track.

An earlier version of the test rig with pads of the felted nickel for brushes was prepared however the felted metal could not be made to wet with the gallium alloy and was too soft to run as a brush material by itself as the surface smeared very quickly blocking the pores and galling, (Fig. 5).

RESULTS AND DISCUSSION

Principal Experimental Results

Data are reported for three major experimental setups, each with a different type of brush. Speed is reported as nominal speed, based upon controller setting and no-load calibration. Current is in rms AC amperes. Contact voltage drop is in rms AC volts per contact. Three sets of data are reported in tables III, IV and V.

TABLE III. TEST DATA FOR CARBON BLOCK BRUSHES RUN ON COPPER PLATED STEEL

| Run | Speed (fps) | Current (amperes) | Voltage (volts) | Load (pounds) | Duration (seconds) |
|-------------------------------|----------------|----------------------|--------------------|------------------|-----------------------|
| C-1 | 31.1 | 100 | 2.0 | 2.75 | 20 |
| C-2 | 16.6 | 100 | 2.1 | 2.75 | 45 |
| C-3 | 10.4 | 110 | 2.15 | 2.75 | 40 |
| C-4 | 10.36 | 115 | 2.15->2.5 | 2.75 | 40 |
| Gallium Alloy Wetted Contact: | | | | | |
| C-5 | 10.36 | 115 | 1.4->2.5 | 2.75 | 40 |

Notes: Run C-4,5 were run long enough to permit examination of system. In C-4,5 the initial contact voltage was reported. Runs C-1,2,3,4,5 were stopped when incandescent wear product was observed. In C-4,5 Initial and final voltage drop were recorded.

TABLE IV. TEST DATA FOR COPPER BRAID BRUSHES RUN ON COPPER
PLATED STEEL WITH GALLIUM ALLOY

| Run | Speed (fps) | Current (amperes) | Voltage (volts) | Load (pounds) | Duration (seconds) |
|------|----------------|----------------------|--------------------|------------------|-----------------------|
| B-1 | 10.4 | 110 | -- | <1 | 20 |
| B-2 | 10.4 | 110 | 0.026 | 5.25 | 83 |
| B-3A | 10.4 | 110 | 0.035 | 5.25 | -- |
| B-3B | 16.6 | 110 | 0.017 | 5.25 | -- |
| B-3C | 16.6 | 110 | 0.020->0.035 | 5.25 | 268 for A,B,C |

Notes: Good coverage of the wear track and good wetting of the braids was observed. Alloy was observed to pool at leading edges of brushes. Alloy was lost by slinging during operation, and appeared to "dry up" on wear track, possibly as the result of oxidation. Wear track rewetted immediately and operation returned to original excellent behavior upon application of fresh liquid alloy in B-3A,B,C.

TABLE V. TEST DATA FOR HYDRODYNAMIC BRUSHES ON COPPER
PLATED STEEL WITH GALLIUM ALLOY

| Run | Speed (fps) | Current (amperes) | Voltage (volts) | Load (pounds) | Duration (seconds) |
|------|----------------|----------------------|--------------------|------------------|-----------------------|
| H-1A | 10.4 | 110 | 0.014 | 2.5 | -- |
| H-1B | 13.6 | 160 | 0.0176 | 2.5 | -- |
| H-1C | 13.6 | 300+ | 0.265 | 2.5 | 330 for A,B,C |

Notes: Ammeter was limited to 300 amperes. Runs were made in one setup with replenishment of liquid alloy. Operation was notably without distress. Runs were stopped by burnout of connector remote from brushes.

Discussion of Results

The carbon brushes were never considered to be serious candidates for high current. They provided a useful vehicle for shakedown, and a comparison standard by which the braids and hydrodynamic fingers could be assessed. The tests did provide a valuable item of data which supported the modified thermoelastic theory of monolithic brushes, and provided a measure of functional hardness during high temperature operation, as well as a measure of contact patch size.

From Holm's theory of constriction resistance, assuming that any contaminant films were primarily carbon, an a-spot diameter z was calculated, and from this a nominal contact pressure was calculated. This was corrected for junction growth, assuming that friction coefficient has approached unity. Constriction resistance is given by:

$$R = r^*/z$$

Where r^* for the material used is resistivity and is 800 ohm-cm. The resistance R was calculated from current and voltage drop.

From Table VI we see that the nominal hardness is near to but above the compressive strength of the carbon, 3530 N/cm². We see also that the gallium lubricant significantly lowered contact resistance until it was removed from the wear track.

TABLE VI. CALCULATED DATA FOR CARBON/COPPER CONTACT

| Run | R (ohms) | z (mm) | P (N/cm ²) | P _{corrected} |
|----------|-------------|-----------|---------------------------|------------------------|
| C-1 | .02 | .40 | 2391 | 5436 |
| C-2 | .021 | .38 | 2650 | 6023 |
| C-3 | .019 | .41 | 2276 | 5174 |
| C-4 | .019->.022 | .43 | 2226 | 5056 |
| C-5 | .012->.022 | .65 | -- | -- |
| <hr/> | | | | |
| Avg.1->4 | .02 | .405 | 2385 | 5420 |

Turning to the copper braid experiments, we found early in the program that the braid wetted much more readily than stainless steel and nickel porous materials we had obtained. Consequently a brush was made by laying up a sandwich of five braids clamped between mild steel jaws. Steel was chosen after an initial experiment with aluminum jaws which exploded soon after the gallium alloy was applied. The nominal contact face was 0.1 in. wide and 0.75 in. long, and was smoothed by abrasion.

Good coverage of the wear track and good wetting of the braids was observed. Contact resistance was approximately one tenth that observed for the carbon. Nominal current density, amperes per square inch, was 1466, as compared with 586 which was used with the carbon.

The brushes ran without distress until the film thinned, and

then a rise of contact resistance was observed to begin. The apparatus was stopped and fresh alloy was laid on the wear track with a syringe. It immediately wet and spread, and performance returned to its initial measures.

The configuration of the apparatus did not permit scavenging thrown-off alloy for reintroduction to the wear track. Only experiments in a suitable geometry will show the true lubricant consumption.

The hydrodynamic fingers showed the most impressive performance. There was no sense of approaching limits of operation. The voltage wave on the oscilloscope was clean and without indications of interruptions or noise. We believe they could have been operated at considerably reduced load, as they were flexible enough to assure full contact. Based upon the nominal contact area they were carrying 2467 amperes/in².



FIGURE 1: Overall view of the turntable rig showing the turntable, the motor and its controller, the brush support arms, and the power supply.

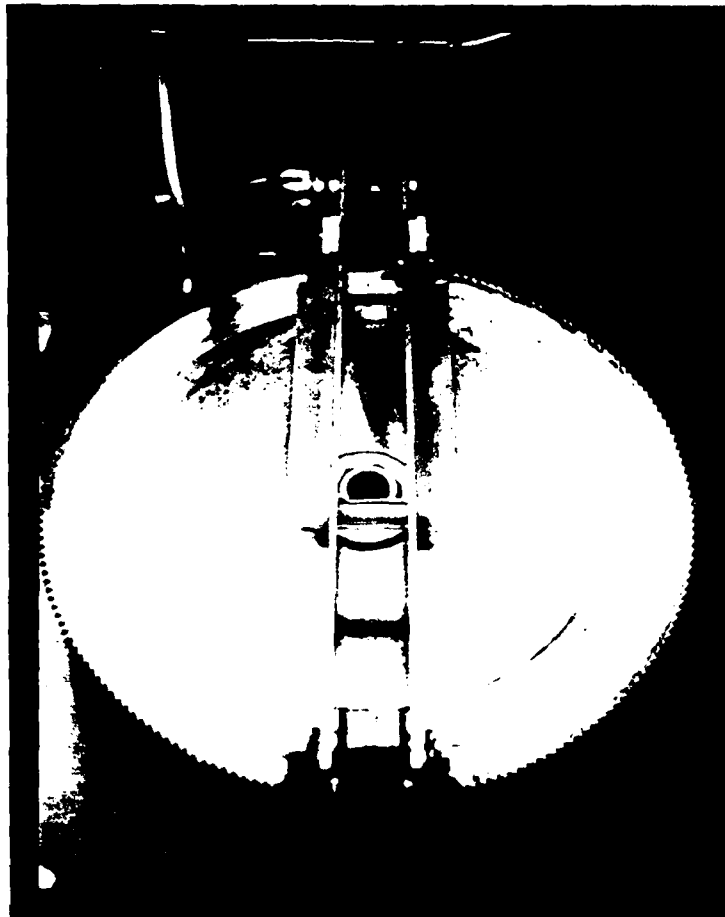


FIGURE 2: The turntable rig in operation showing the gallium alloy track, and the friction measurement equipment.



FIGURE 3: Detail of the turntable showing the method of loading the brushes using a spring dynamometer.

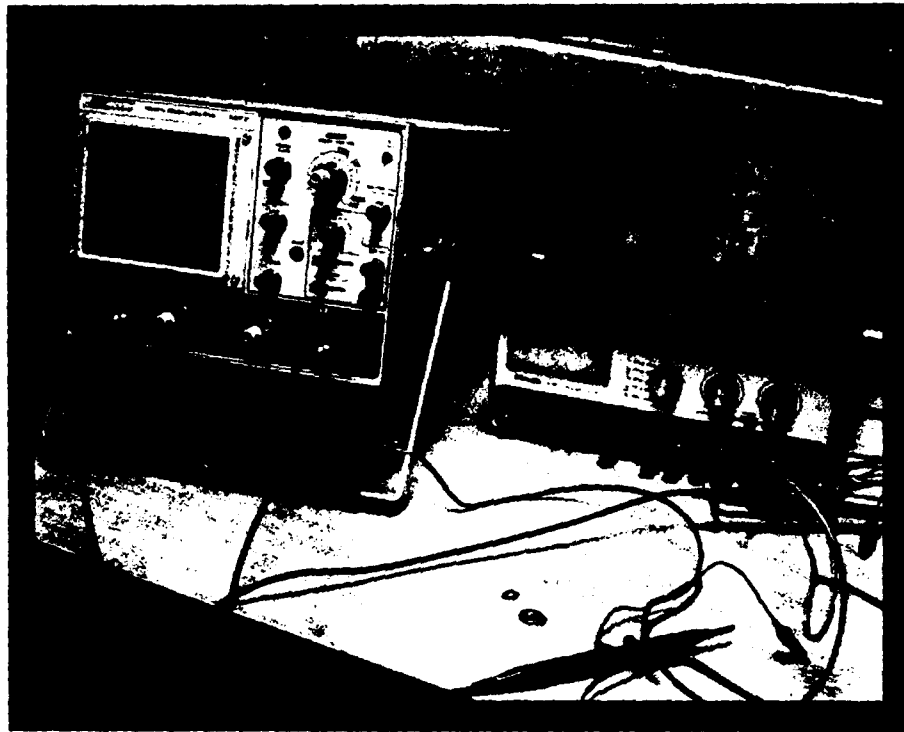


FIGURE 4: Photograph of the oscilloscope and power supply for the proximeters.

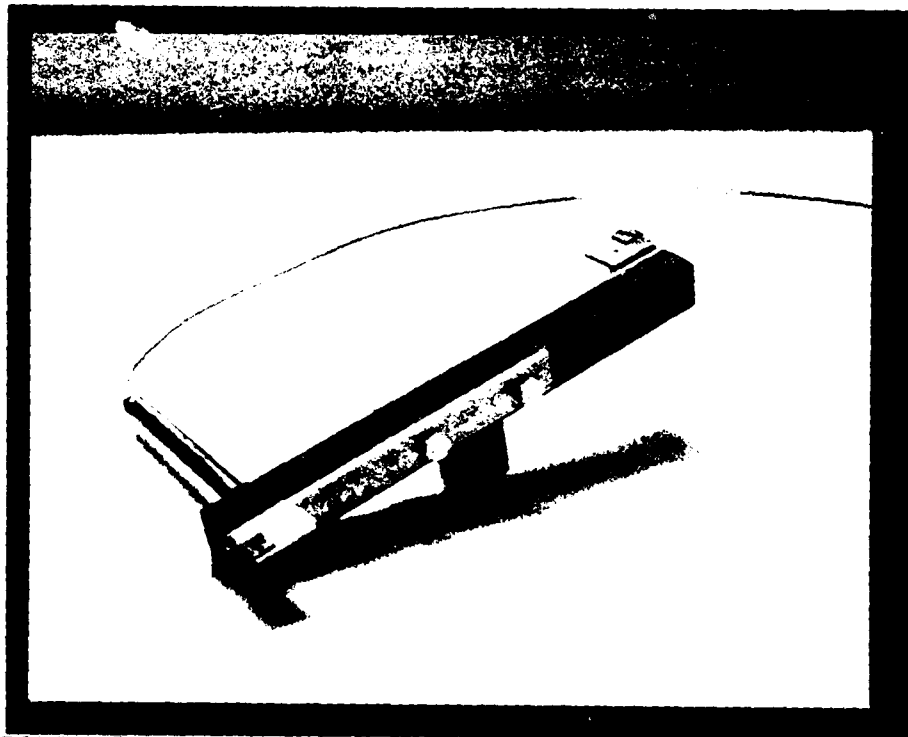


FIGURE 5: An early version of the brush mounting assembly from the turntable rig showing the proximeter mounting. The brush pads are made from the felted nickel which demonstrated poor wetting and structural properties.

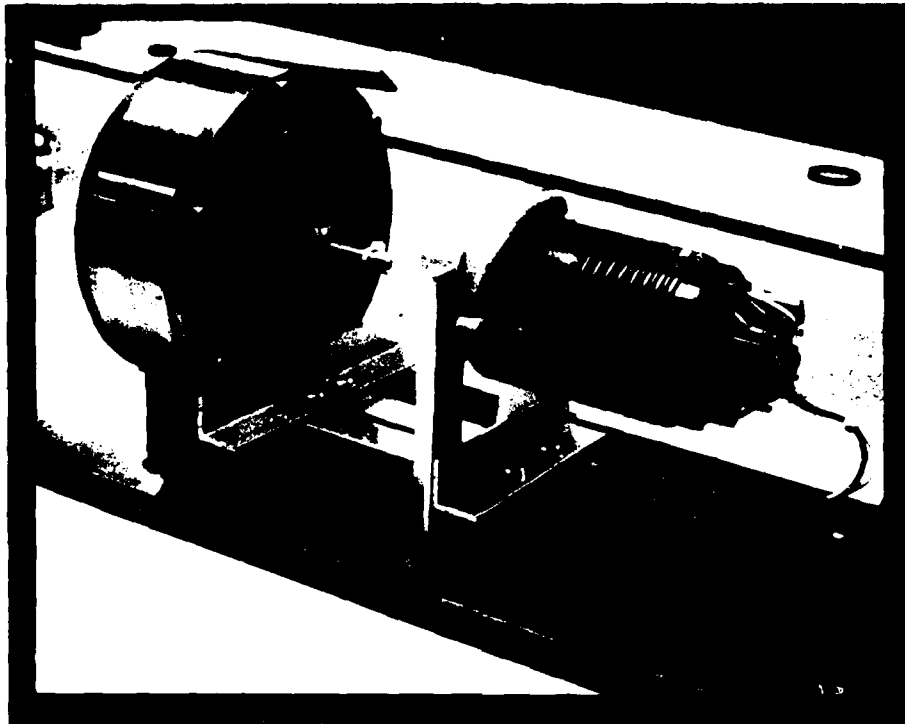


FIGURE 6: Photograph of the long duration test homopolar device. This device is being instrumented for computer monitoring and control.



FIGURE 7: This photograph shows the rewetting of a dried out gallium alloy contact track.

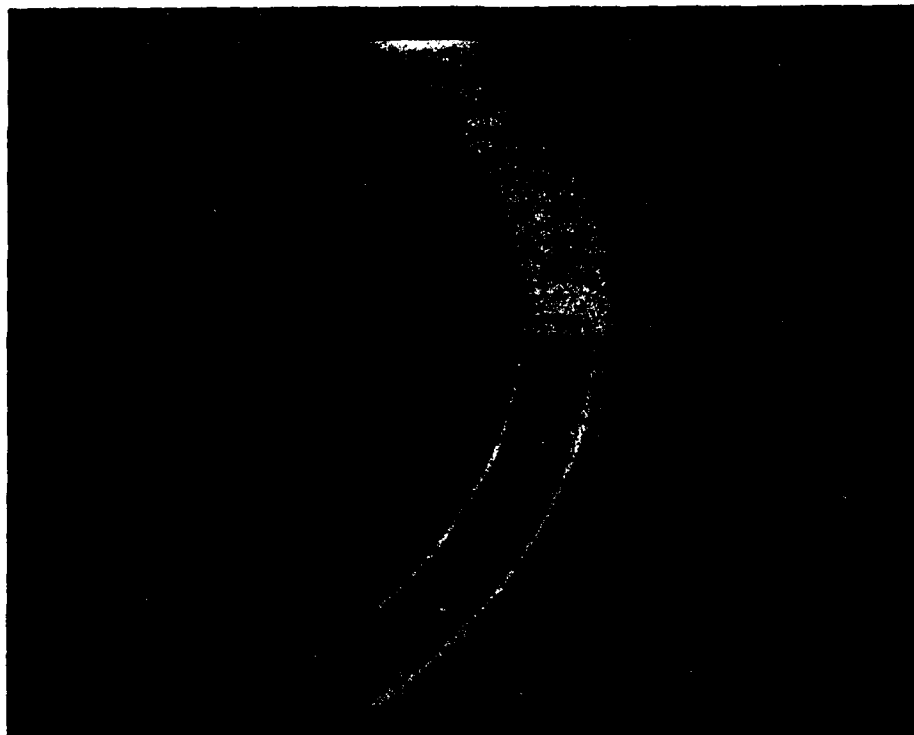


FIGURE 8: This is the wear track of the copper finger brushes after 5.5 minutes of operation at almost 2500 amps/in². The track showed no dulling or drying in the argon atmosphere.



FIGURE 9: The wear track in this photograph shows the damage to the specimen from the gallium alloy-aluminum reaction. This reaction was explosive.

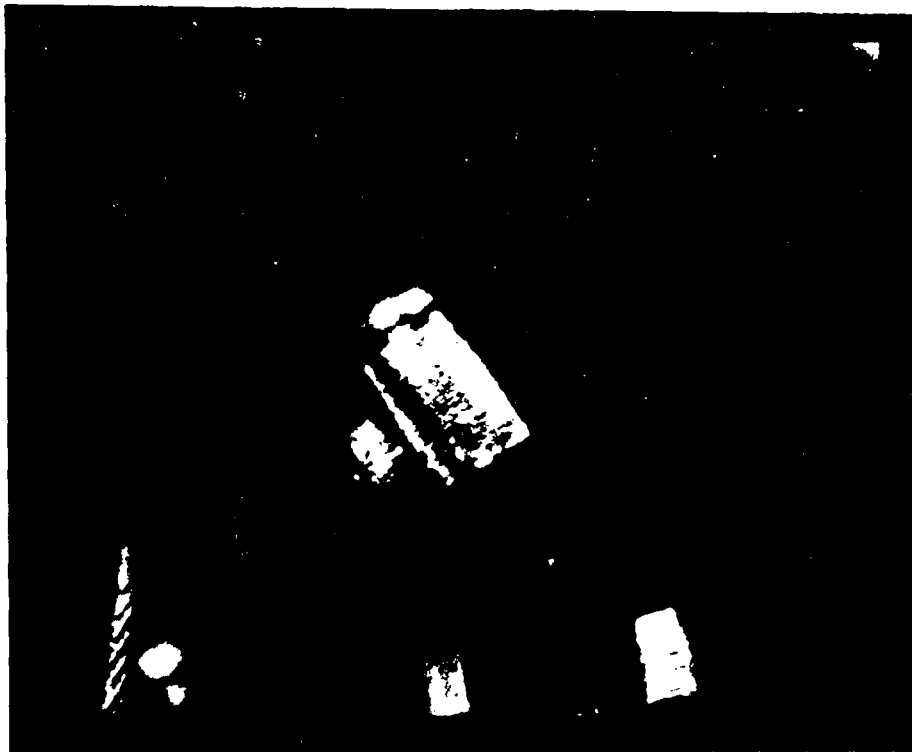


FIGURE 10: Brush fabricated from copper desoldering braid after use with the gallium alloy and several wetting and dry out cycles.



FIGURE 11: Comparison of graphite brushes before and after operation in air on gallium alloy. For scale purposes 1/4 inch cap screws are depicted.

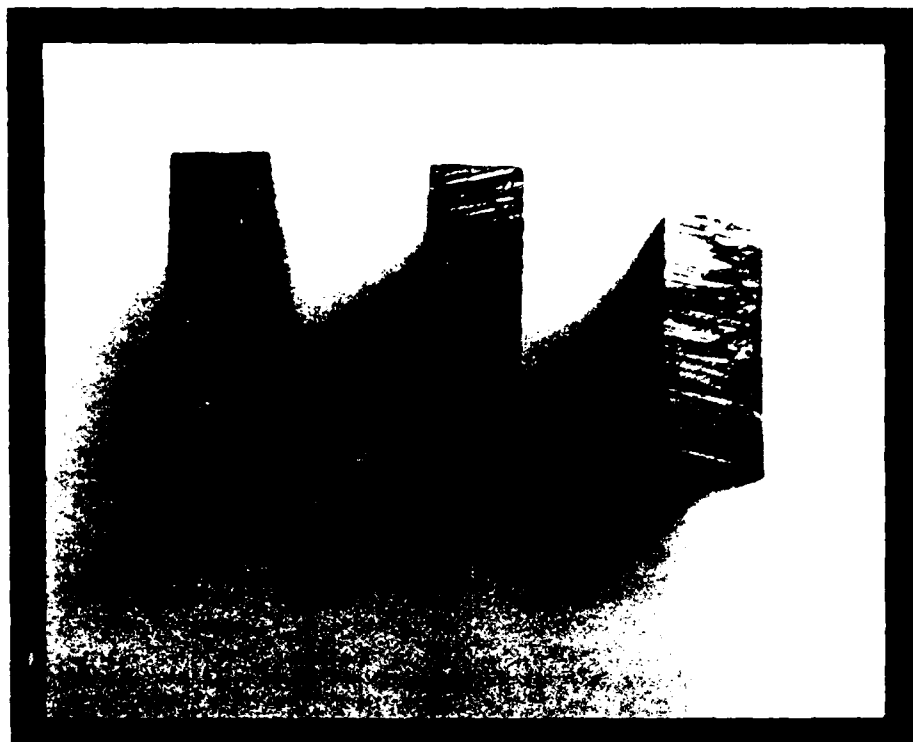


FIGURE 12: Comparison photograph of graphite brushes run in air, from left to right new, dry, gallium wetted all after testing.



FIGURE 13: Copper finger brushes after 5.5 minutes of operation on gallium alloy in argon at 2467 amps/in².

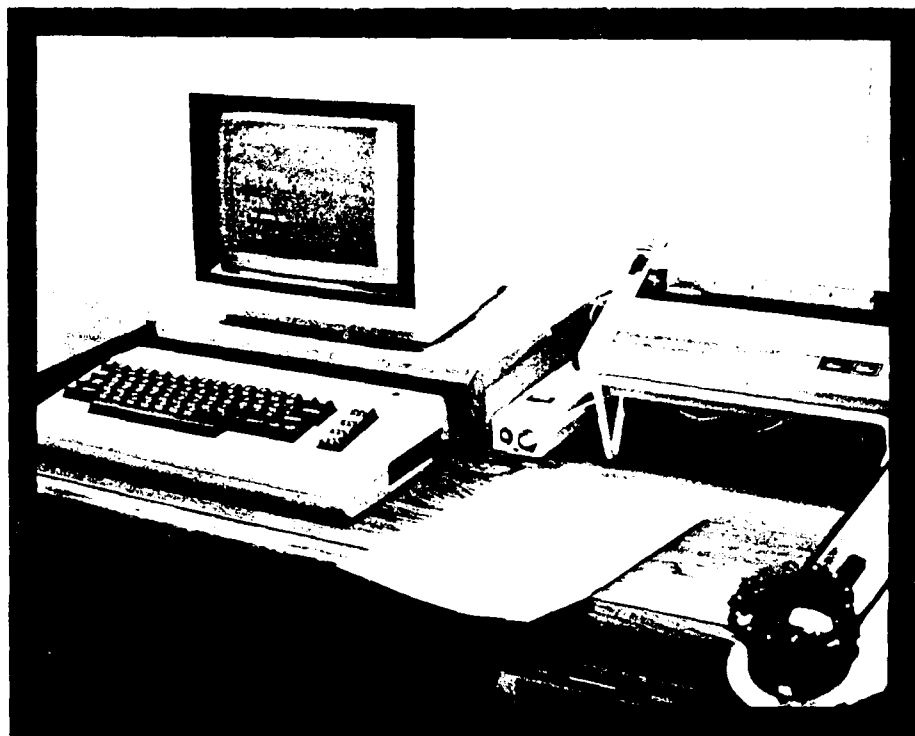


FIGURE 14: The computer data logging system in operation, this systems operates both input and output at 100 cycles/second. The A/D controller is a National Semiconductor ADC0817CCN 8 bit converter.

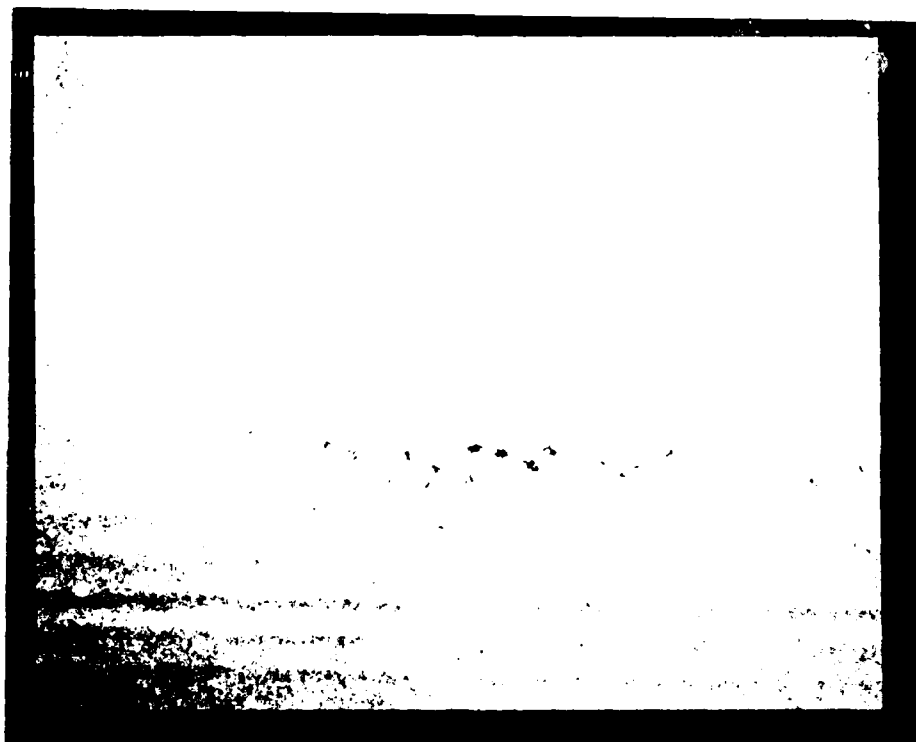


FIGURE 15: This shows the liquid phase of the gallium alloy after being slung from the turntable.

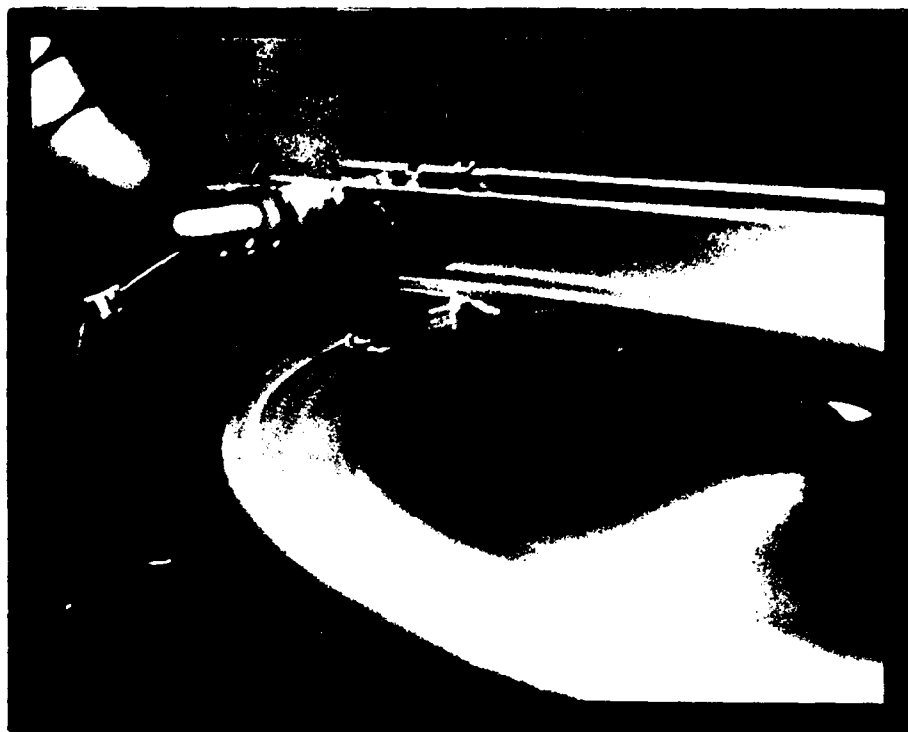


FIGURE 16: Leading edge of the graphite brush showing the bead of liquid alloy against the brush face.

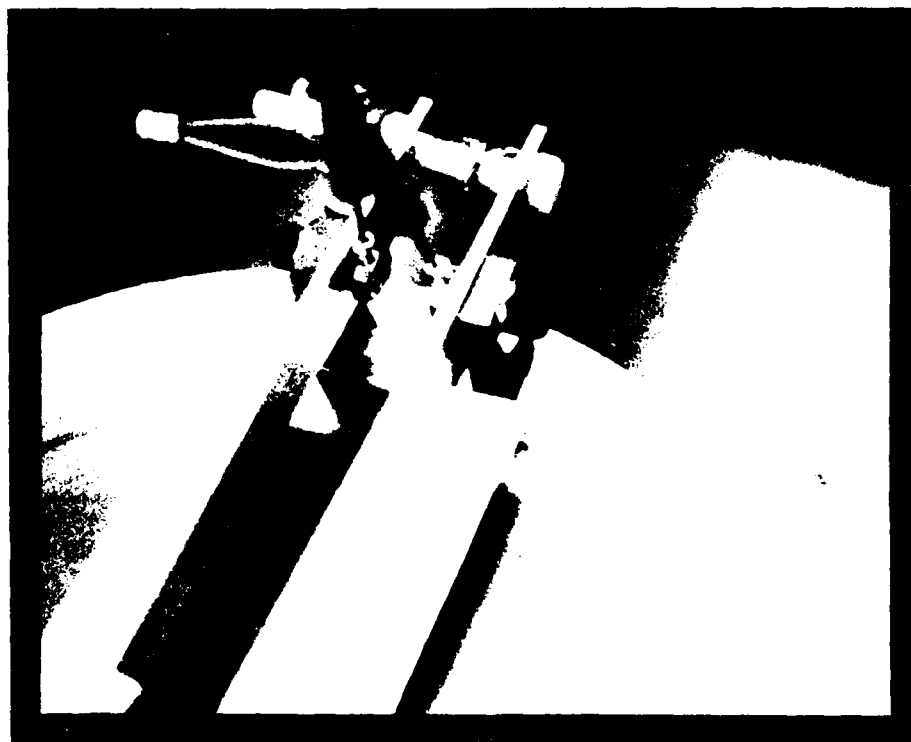


FIGURE 17: Wear track of the graphite brush on the liquid-phase gallium alloy demonstrating the clean breakaway from the brush.



FIGURE 18: Application of the gallium alloy to the copper plated surface of the turntable was facilitated by ready wetting properties of the gallium alloy on the copper and could be spread with a simple plastic instrument.

FIGURE 19 MAXIMUM TESTED CURRENT DENSITY

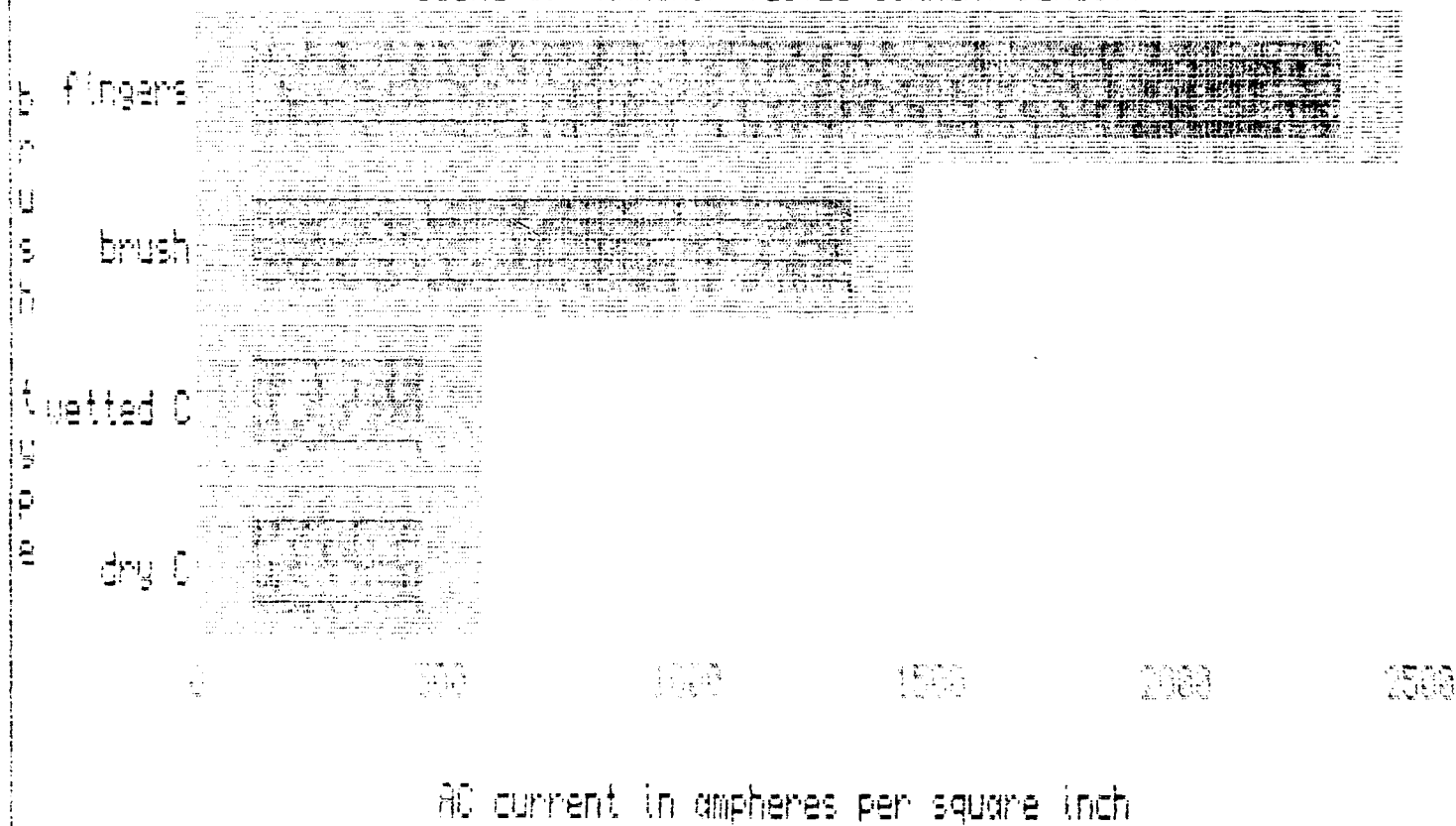


FIGURE 20 CONTACT RESISTANCE

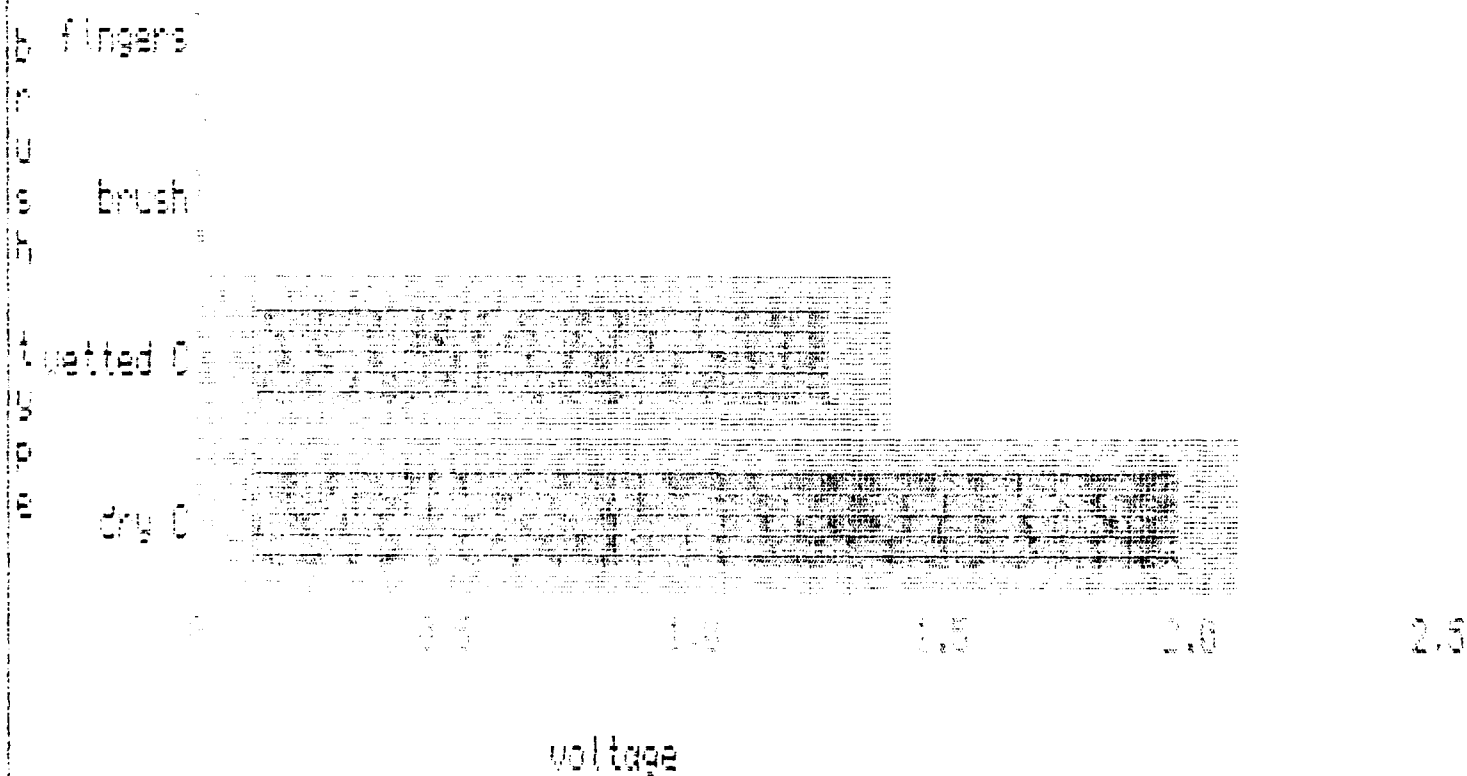
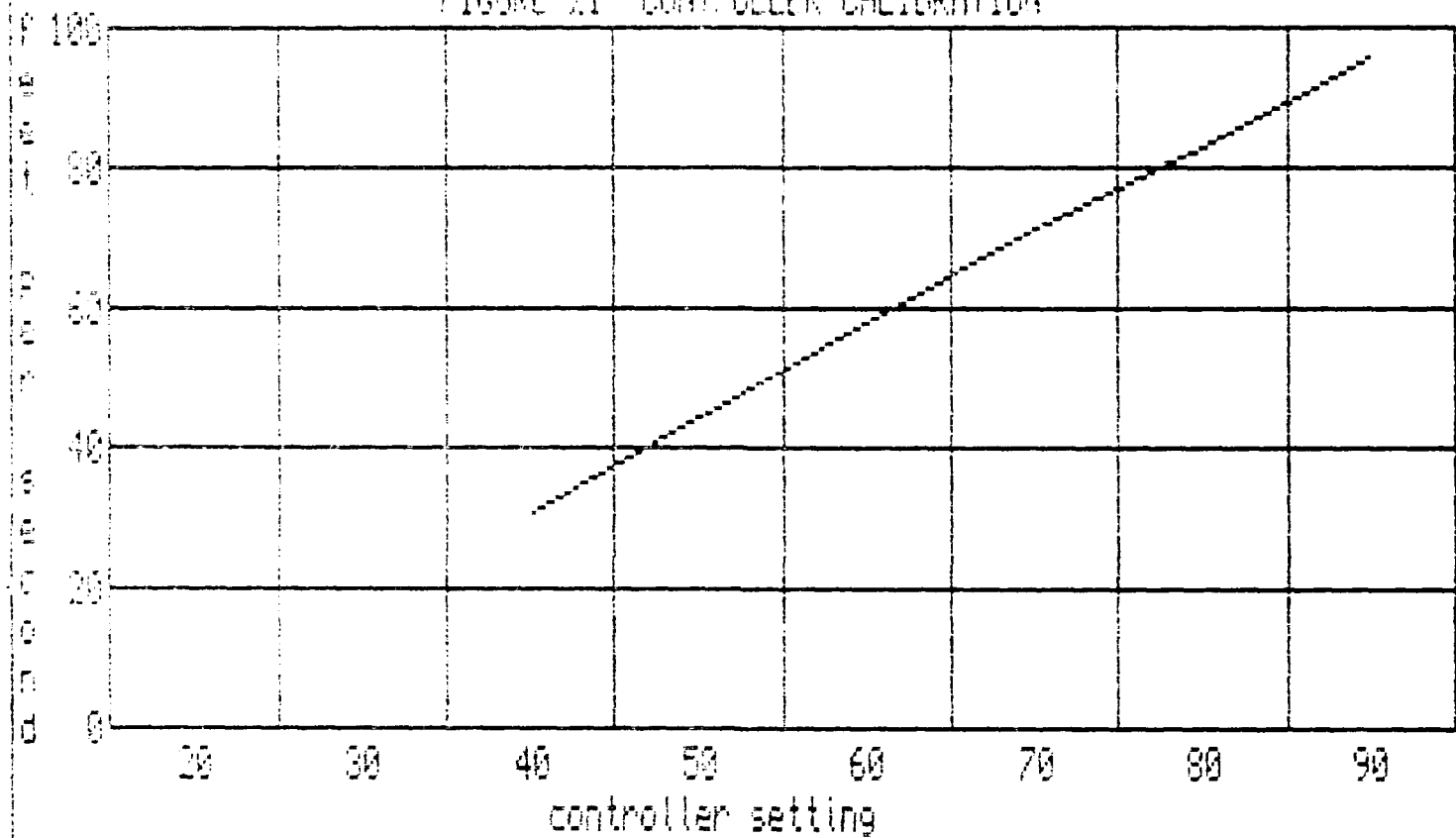


FIGURE 21 CONTROLLER CALIBRATION



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